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MATING AND OPTIMIZATION PARAMETERS FOR HIGH-TEMPERATURE LIQUID METAL WETTING ON SOLID SUBSTRATES

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14. ABSTRACT
The main goal of the research is to determine the mating and optimization parameters for the wetting of liquid metal coolants, such as sodium (Na) or lithium (Li), onto various solid surfaces by experimentally measuring the wetting angles and theoretically examining the surface energies. The wetting angle is optically measured using a microscopic goniometer and digital imaging analysis. Examination of the mating and optimization wetting parameters can provide a valuable data base of design parameters for high-temperature heat pipes using liquid metal coolants. Due to budget constraints, this in-house effort was cancelled prior to the start of testing; therefore, only the test chamber design and preparations are discussed in this report.

15. SUBJECT TERMS
liquid metal wetting, heat pipe, wetting angles, surface energies, goniometer, capillary transport

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NOMENCLATURE

AF	Air Force
AFRL	Air Force Research Laboratory
atm	atmospheres
°C	degree Celsius
CCD	charge-coupled device
Cu	copper
CT	capillary transport
Dia	diameter
DC	direct current
FIRST	Facility for Innovative Research in Structures Technology
FPS	frames per second
Li	lithium
Mil-Std	military standard
Mo	molybdenum
Na	sodium
Nb	niobium
NI	National Instruments
PC	personal computer
PCI	peripheral component interconnect
PXI	PCI extensions for instrumentation
RQ	Aerospace Systems Directorate
SOP	standard operating procedure
SS	stainless steel
UV	ultra-violet
V	volts
WPAFB	Wright-Patterson Air Force Base

1.0 SUMMARY

The main goal of this research program is to determine the mating and optimization parameters for the wetting of liquid metal coolants, such as sodium (Na) or lithium (Li), onto various solid surfaces by experimentally measuring the wetting angles and theoretically examining the surface energies. The wetting angle is optically measured using a microscopic goniometer and digital imaging analysis. Examination of the mating and optimization wetting parameters can provide a valuable database of design parameters for high temperature heat pipes using liquid metal coolants.

The experimental system includes an inert-gas (Argon) purged smelting section that provides a single drop of liquid metal through a hypodermic needle into the lower test section which is also in an inert-gas purged environment. The lower test section consists of the test sample substrates, which are made of Niobium (Nb), Molybdenum (Mo), or their alloys, a plate heater to maintain the test sample at a desired temperature, and thermocouple probes. Both the upper smelting section and the lower test section have been designed, fabricated, and test-proofed at the Facility for Innovative Research in Structures Technologies (FIRST) Laboratory at Wright-Patterson Air Force Base (WPAFB). The whole experimental setup has been successfully implemented to launch the wetting angle measurements and all of the test sample substrates have been prepared to accommodate the planned test condition matrix. Unfortunately, the research program was cancelled as of April 2013 prior to the collection of any test data. Therefore, this report will only discuss the design and construction of the experimental setup and the matrix of tests that would have been performed if this project had continued through completion.

2.0 INTRODUCTION

The objective of this effort is to measure the detailed physics associated with the micro-scale heat and mass transport phenomena of liquid metal coolants of Capillary Transport systems (CT). It is intended that the product of this effort will enhance understanding of capillary wicking thermal transport systems operating in a high temperature environment. Success in accomplishing the program objective would contribute to enhancing the extreme cooling of shock wave heat from in-flight conditions experienced by supersonic/hypersonic vehicles.

The mating and optimization parameters will include the fluidic properties, the material properties, and the operational properties. The fluid properties include the liquid metal surface tension, viscosity, type of surfactants (if applicable), and compositions (in cases where mixtures are used). The material properties of interest are the wicking dimensions and structure, the wicking alloy type, and the surface finish. The operational properties of note are the temperature of the liquid metal, temperature of the wicking material, and the environmental conditions (such as vapor or inert gas).

Pure and oxidation-free liquid Na and Li will be refined in a vacuum or inert gas environment using a uniquely designed smelting vessel. The liquid Sodium and/or Lithium will then be extracted using a high temperature injection needle to form a drop onto the test wicking surface. The wetting angle will be optically measured using microscopic goniometer and digital imaging analysis software. The surface energy coefficient will be measured as well. Measurements will be repeated for different combinations of the aforementioned mating and optimization parameters.

The measured wetting angle data will be imported as design parameters into a valuable data-base for high-temperature heat pipes using liquid metal coolants. The data will also will be used to confirm the theory that the free electrons within liquid metals enhance heat transfer. This research will also confirm current prediction models of surface energy and surface tension coefficients of the liquid metals (Na, Li) under different mating and optimization parameters.

Section 3 presents detailed design and construction of the smelting chamber, the heated test chamber, and their implementation at the Air Force Research Laboratory's (AFRL) FIRST Laboratory. Section 4 summarizes some preliminary efforts that were devoted to initiate the experimental test matrices.

3.0 EXPERIMENTAL SETUP

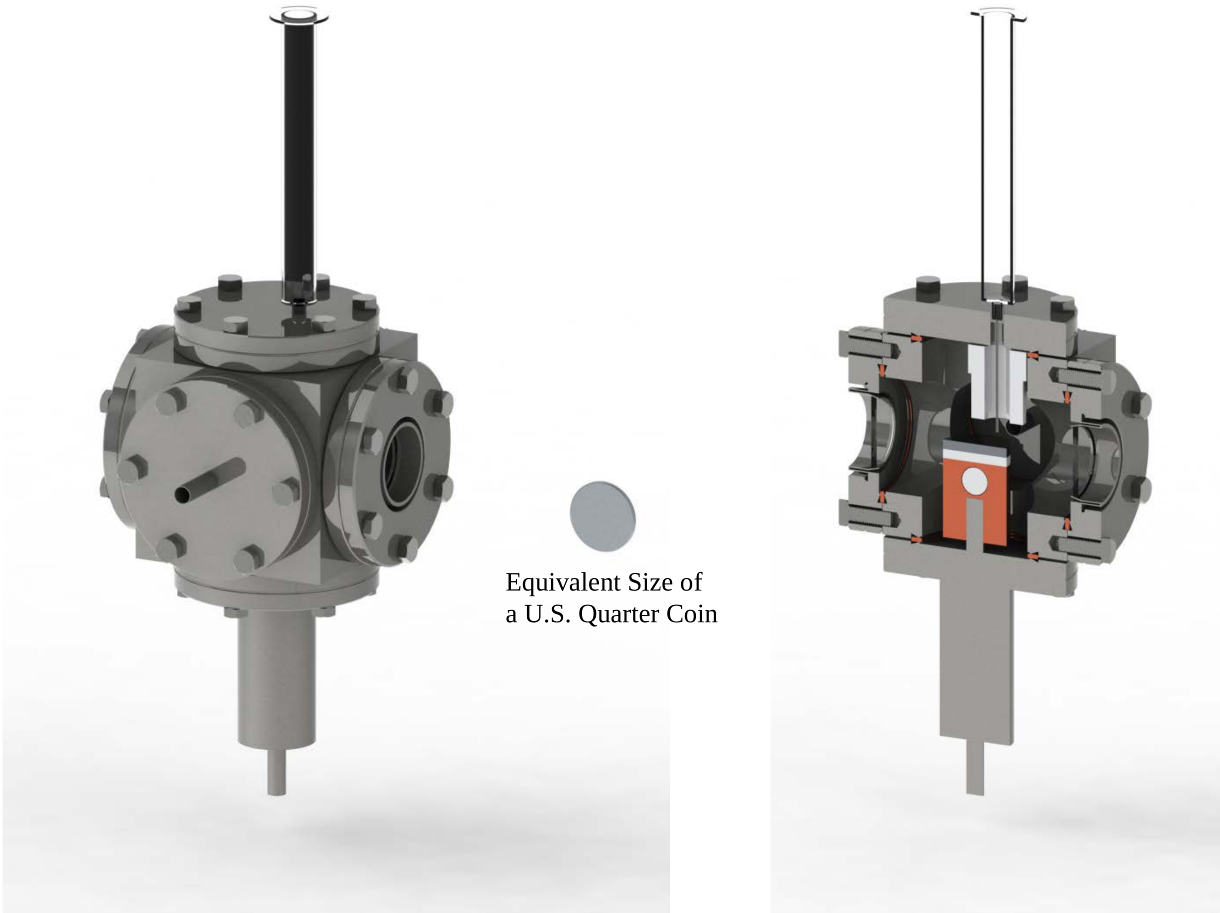
Section 3.1 presents detailed design and construction of the test chamber that consists of the upper smelting section and the lower heated test section. Section 3.2 presents the list of all of the parts and also describes the assembly of the test chamber. Finally, Section 3.3 describes the induction coil heating system and the goniometric wetting characterization system.

3.1 Test Chamber Design and Construction

To perform this unique set of experiments a custom test chamber was designed. Figure 1a and 1b show the test chamber design and its cut-away view, respectively. The test chamber consists of two sections: the upper smelting section to allow a single drop of pure melted liquid metal through the hypodermic needle, and the lower heated test section to heat and hold a test sample.

More detailed descriptions for individual parts are presented in Figure 2. Note that the syringe needle is off-center so that the rotating base can accommodate multiple drops before re-filling the syringe with the metal sample. A coin-shaped heater is used to heat the test sample. The test sample is placed on top of the Copper (Cu) block, which is heated by a cylindrical heater element. The Ferro-magnetic fluid rotary feed-through turns the test sample to accommodate multiple liquid metal drops. The ultraviolet (UV) sapphire view ports allow optical access under high-temperature operations. The entire chamber will be filled with inert argon gas to avoid oxidation of the liquid metal sample surfaces.

Figure 3 shows a schematic cutaway view of the final assembly of the experimental setup. The test chamber is placed on a supporting base, which is placed on a jack for precise height adjustment.



(a) **(b)**

Figure 1 (a) Test Chamber Design and (b) Cutaway View

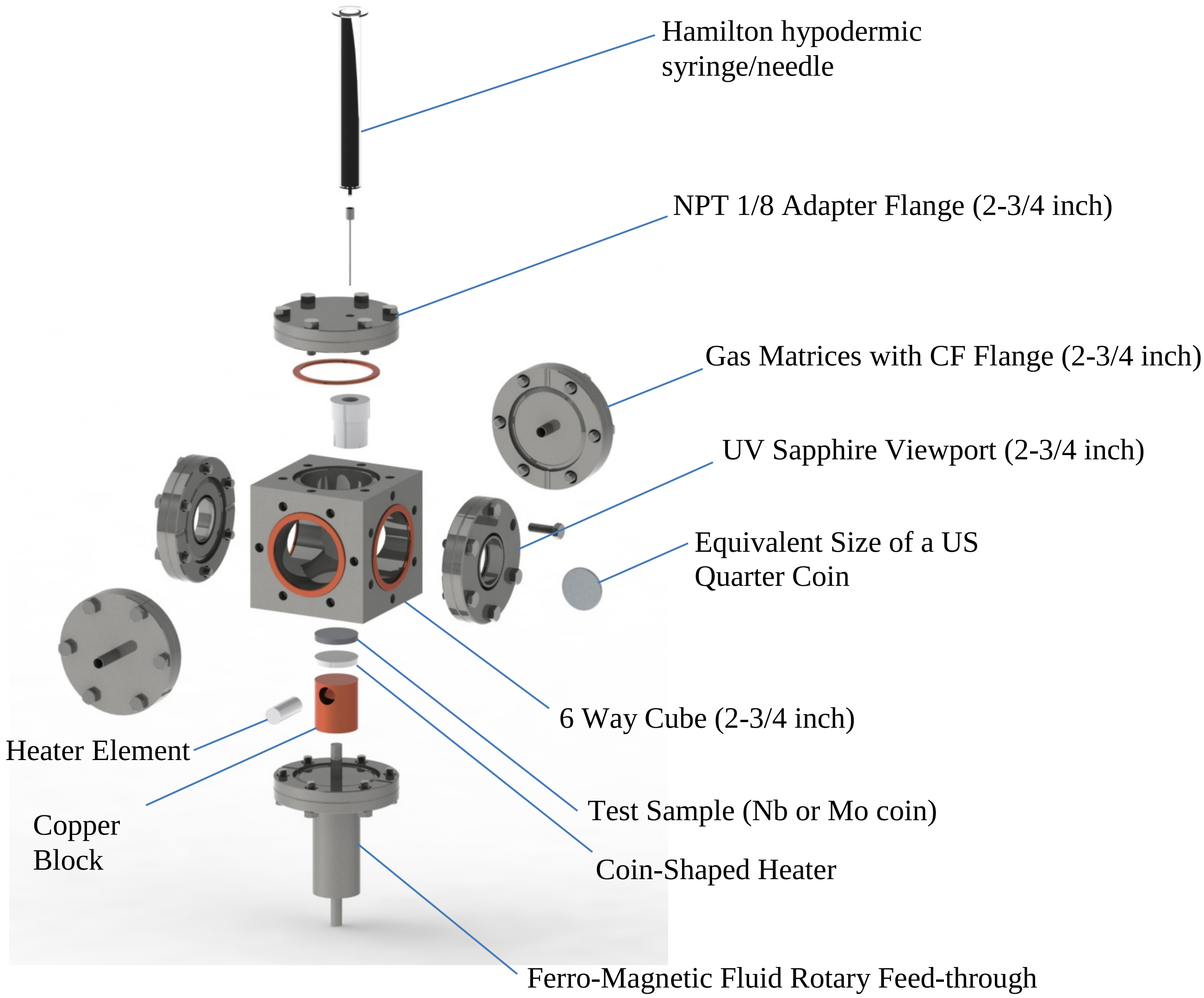


Figure 2 Test Chamber Components

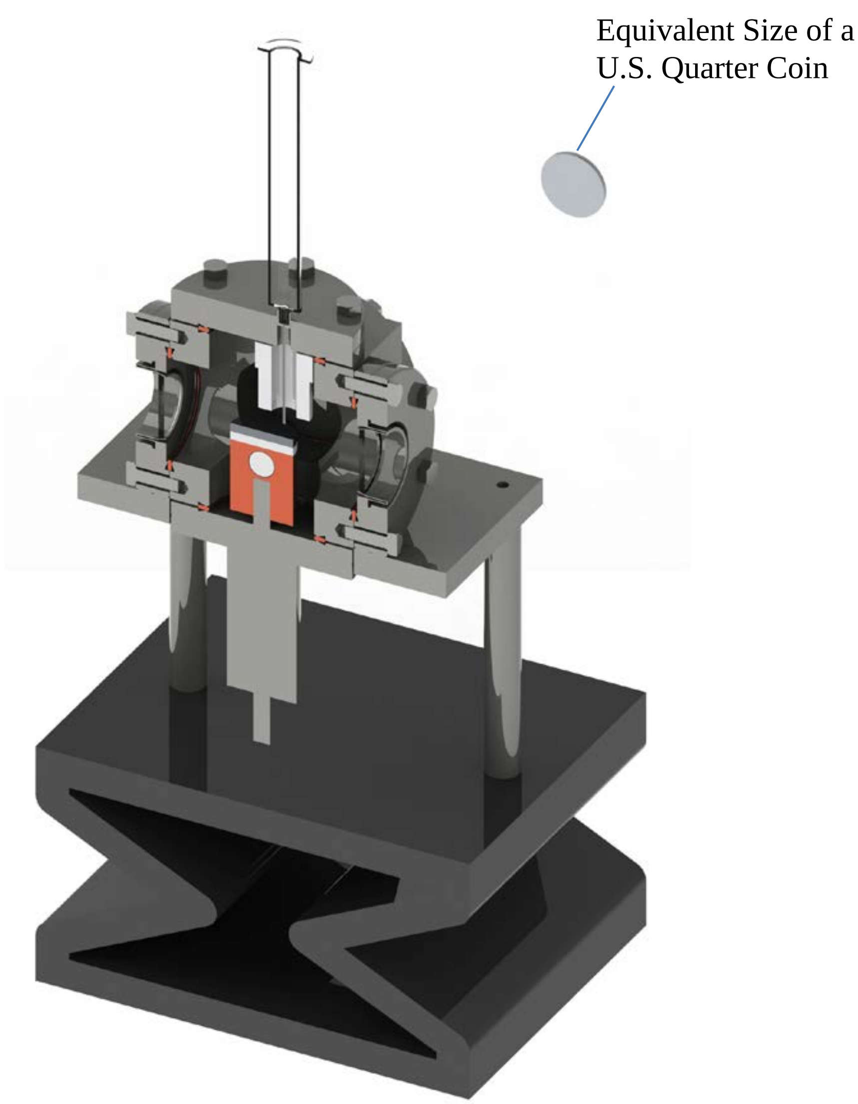


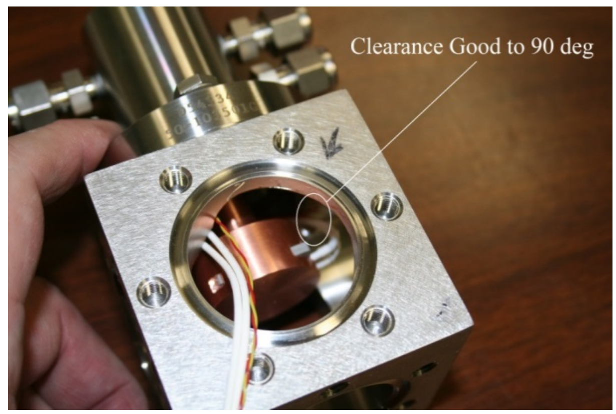
Figure 3 Cutaway View of Final Assembly

3.2 Test Chamber Parts and Setup

Figures 4-a to 4-d show the detailed parts for the test section assembly including: (a) the stainless steel test section parts, (b) the hypodermic needle syringe to be used for the smelting section, (c) the Ferro-magnetic fluid rotary feed-through unit, and (d) the Copper block with the heater element inserted.

(a) **Stainless Steel Parts**

(b) Hypodermic Syringe



(c) Rotary Feed-Through Unit

(d) Cu Block and Heat Element

Figure 4 Test Chamber Assembly Parts

Testing will be conducted in a 24- by 24- by 48-inch vapor containment enclosure connected by a 6-inch diameter flexible hose to a negative pressure exhaust system, as shown in Figure 5. The enclosure is fabricated from a Unistrut© frame and clear acrylic (Plexiglas) panels. One side of the enclosed area can be opened to access the test vessel. The free-standing test vessel will be set in a stainless steel spill pan. It is highly unlikely that the test vessel will fail, however if the vessel physically fails during testing, any hazardous vapors released will be contained by the enclosure and removed by the exhaust vent. Any solids or liquids will fall into the spill pan. A class D fire extinguisher will be available nearby.

Figure 5 Vapor Containment Enclosure

3.3 Equipment and Instrumentation

Figure 6 shows an Ambrell© induction coil heating system that will be used to heat and melt the solid metal ingot placed inside the glass syringe. Figure 7 shows the goniometer system to measure the contact angles and drop profiles. The system also includes digital image analysis tools to determine the detailed wetting parameters.

The data acquisition system will be personal computer (PC)-based using custom LabView© application written for this test setup. The instrumentation interface is a National Instruments PXI-1042 chassis with the following interface cards installed:

- One (1) PXI-8336 fiber-optic communication module.
- One (1) PXI-GPIB communication module.
- One (1) PXI-4351 temperature/voltage input module.
- Two (2) PXI-6229 multifunction modules.
- One (1) PXI-1428 image acquisition module.
- One (1) NI TBX-68T thermocouple/voltage input terminal block.
- Four (4) NI SCB-68 instrumentation terminal breakout block.

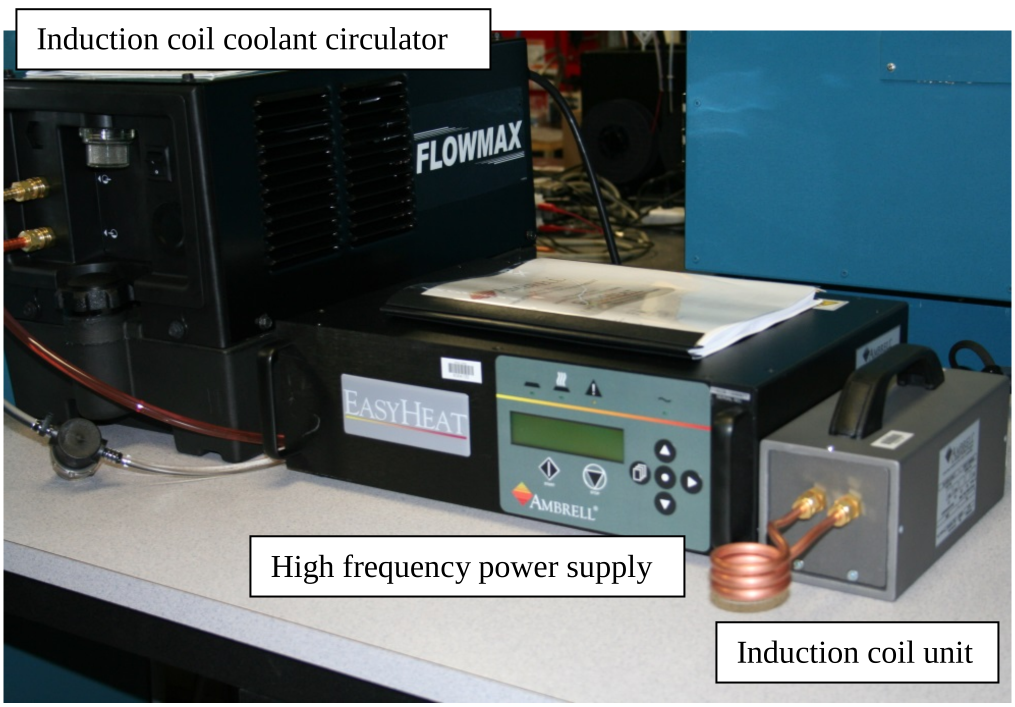
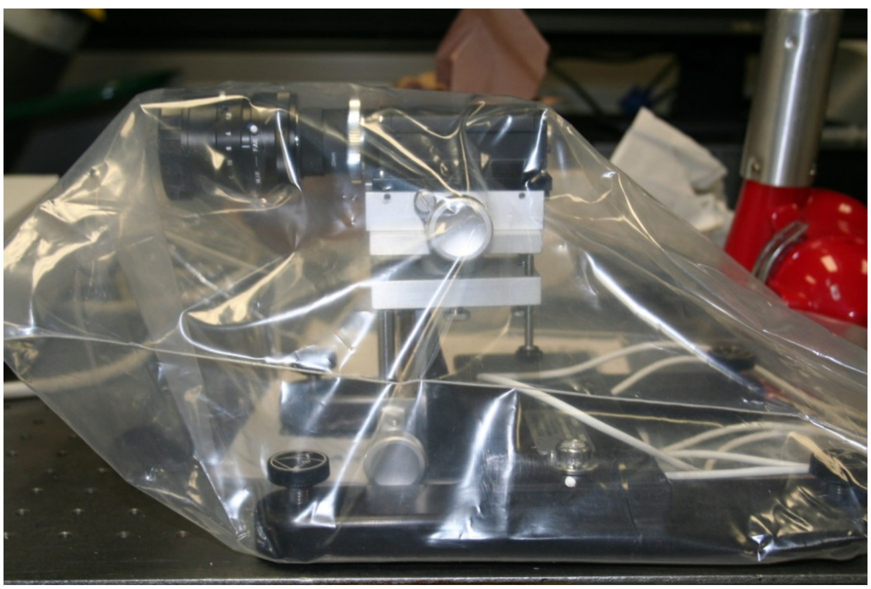


Figure 6 Induction Coil Heating System

(a) **Parallel Incident Lighting Unit**



(b) **Telescopic Charge-Coupled Device (CCD) Detector Unit**

Figure 7 Goniometer System

4.0 PRELIMINARY EFFORTS

Preliminary efforts have been primarily for the data acquisition protocols, safety precautions, and anticipated test procedure. More detailed descriptions are itemized in the following sections.

4.1 Data Acquisition

The following data will be recorded:

- Parameters to be Measured:
    - Goniometer instrumentation/video camera: a minimum of 61 frames per second (FPS) – Software controlled.
    - Temperature utilizing type "K" thermocouple probes.
    - Heater shunt and power supply voltage utilizing differential voltage sensors.
    - Microscopic video camera images utilizing the PXI-1428 communication port.
- Measurement Ranges:
    - Temperatures: 0 – (25% below boiling temperature of particular metal) °C.
    - Heater voltage drop: 0 to 20 V dc range.
- Accuracy and Resolution Required:
    - Temperatures: ±1 °C.
    - Heater shunt voltage: ±0.01 V dc.
    - Heater power supply voltage: ±0.1 V dc.
- Sample Rate:
    - Temperatures: 1 sample/s.
    - Heater voltages: 1 sample/s.
    - Microscopic video camera: minimum 61 FPS.
- Parameters to be Calculated:
    - Heater current and voltage for test vessel heater power supply voltage control.

4.2 Test Matrix

A preliminary test matrix was developed and is shown in Table 1. Test runs would be performed with varying liquid metal, substrate, and mesh materials. The geometry of the mesh and the test temperatures would also be altered to fully characterize the different CT systems. Each run would consist of three drops of liquid metal on the substrate material. The measured data would have been compiled in a database used to optimize CT system designs for high heat flux applications.

Table 1 Preliminary Test Matrix

Liquid Metal Test Matrix										
Sodium	Lithium		SUBSTRATE MATERIAL							
			Molybdenum	Niobium	NickelVac L-605	Haynes 230	Molybdenum Grooved	Niobium Grooved	NickelVac L-605 Grooved	Haynes 230 Grooved
MESH TYPE	Molybdenum	50 x 50 Plain, Wire Dia 0.002"	300C, 450C, 600C	300C, 450C, 600C			300C, 450C, 600C	300C, 450C, 600C		
		60 x 60 Plain, Wire Dia 0.004"	300C, 450C, 600C	300C, 450C, 600C			300C, 450C, 600C	300C, 450C, 600C		
		100 x 100 Plain, Wire Dia 0.001"	300C, 450C, 600C	300C, 450C, 600C			300C, 450C, 600C	300C, 450C, 600C		
		165 x 165 Twill, Wire Dia 0.002"	300C, 450C, 600C	300C, 450C, 600C			300C, 450C, 600C	300C, 450C, 600C		
	S.S 304	180 x 180 Twill, Wire Dia 0.0025"			200C, 300C, 400C	200C, 300C, 400C			200C, 300C, 400C	200C, 300C, 400C
	Haynes 25	100 x 100 Plain, Wire Dia 0.0042			200C, 300C, 400C	200C, 300C, 400C			200C, 300C, 400C	200C, 300C, 400C
	No Mesh		300C, 450C, 600C	300C, 450C, 600C	200C, 300C, 400C	200C, 300C, 400C	300C, 450C, 600C	300C, 450C, 600C	200C, 300C, 400C	200C, 300C, 400C

Each Test Consists of 3 Drops on the Substrate Surface.

Test conducted in argon environment. Pressure = 1atm

4.3 Test Procedures

System Checkout

1. Turn on power to all equipment except the vacuum or gas pump.
2. Allow all equipment to reach ambient operating temperature.
3. Initialize the data acquisition system.
4. Turn on exhaust vent and confirm proper operation.
5. Check all sensor readings for proper ambient values.

Characteristic Test

6. Turn on the vacuum pump to lower the chamber pressure.
7. Shut off the vacuum pump when a desired vacuum is reached.

8. Purge the chamber with pure argon gas supplied from the pressurized bottle.
9. Check the oxygen sensor reading for the oxygen content remaining in the chamber.
10. Repeat steps 6to 9 until a tolerable oxygen level is reached.
11. Set the desired test vessel initial heater wattage.
12. Monitor all instrumentation and sensors for safe operating ranges.
13. Wait until working media has reached the desired state (steady-state is reached when all test vessel thermocouples do not change more than 2 °C/h).
14. Energize the test vessel evaporator heater to the selected wattage (test article two only).
15. Monitor all instrumentation and sensors for safe operating ranges.
16. Wait until all test vessel thermocouples have reached steady-state (steady-state is reached when all test vessel thermocouples do not change more than 2 °C/h) (test article two only).
17. Record all applicable test data including video images of the meniscus.
18. Signal the data acquisition program that it is time to advance to the next wattage level.

Experiment Shut Down

19. Turn off the test vessel heater power supply.
20. Wait until all thermocouples read < 40°C.
21. Disconnect vessel instrumentation from acquisition system.
22. Power down goniometer.

4.4 Safety

Safety documents covering hazard analysis, operating procedures, prevention response and safety permit are prepared and submitted for approval prior to testing. These documents are as follows:

- AF Form 813 (Request For Environmental Impact Analysis)
    - Capillary Transport specific document is signed and on file.
- Standard Operating Procedure (SOP)
    - The standard operating procedure addresses all test procedures and safety issues involved during normal test conditions. All SOPs are posted and used as a checklist by the engineer and technician during testing.
- Site-Specific Spill Prevention Response Plan
    - All spill prevention response procedures for hazardous materials are identified and documented in the response plan and posted at the test site.
- Test Safety Hazard Analysis
    - This document details all specific hazards per Mil-Std-882D. The approved hazard analysis is posted at the test site.
- AFRL Form 5 Safety Permit
    - This document has been prepared and submitted to the program supervisor and Aerospace Systems Directorate (RQ) system safety manager for coordination and approval. The approved safety permit has been posted at the test site.

5.0 CONCLUSIONS

Although no experimental data was gathered, the design of the test chamber and development of the test matrix were successful. The completion of these two tasks would have enabled the generation of a database containing many design parameters useful in optimizing high temperature heat pipe systems. The test chamber was not assembled prior to the cancellation of this research project, but the design of the chamber would have enabled the program to meet its technical objectives. The preliminary work that is documented in this report covers the steps needed to build the test chamber and perform the required test runs to generate data that will enable liquid metal heat pipe design optimization.

6.0 REFERENCES

[1] Pratt, M., David, Kihm, D., Kenneth, *High-Temperature Liquid Metal Transport Physics of Capillary Pumping Heat Transport System (CPHTS) Research*, AFRL Report AFRL-RB-WP-TP-2009-3230, October 2009.

[2] Mikus, E., Ryan, Kihm, D., Kenneth, *High-Temperature Liquid Metal Transport Physics of Capillary Pumping Heat Transport System (CPHTS) Research*, AFRL Report AFRL-RB-WP-TP-2012-0162, April 2012.